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Glass Transition Temperatures of Liquid Prepolymers Obtained by Thermal Penetrometry

Thermal penetrometry is an experimental technique for detecting the temperature at which a frozen prepolymer becomes soft enough to be pierced by a weighted penetrometer needle; the temperature at which this occurs is called the penetration temperature, T_p . In the vicinity of the glass transition temperature there is a very large decrease in shear modulus; below the glass transition temperature, the sample is hard, often brittle, and resists deformation of any kind. Above the glass transition temperature, the sample is either soft and rubbery or else a viscous liquid which can be easily deformed. A weighted needle does not penetrate the glass formed by a liquid copolymer sample below the glass temperature, but it does so easily and rapidly at higher temperatures.

The apparatus used to obtain penetration temperatures can be set up largely from standard parts; thus, the penetrometer needle is of the type specified by ASTM Method D-5, weight 2.5 g, and it is attached to a freely moving vertical rod conforming to the requirements of the same standard method. The shaft has a weight of 47.5 g, and additional weights may be loaded on top of it; a weight of 150 g performs well with liquid copolymers. In order to detect and measure needle motion, a fixed collar on the vertical shaft holding the penetrometer needle can be made to bear on an arm which drives a pointer through a rack and pinion combination; the pointer scale should permit estimates of needle position to the nearest 0.02 mm. Alternatively, the collar on the vertical arm of the penetrometer can be rigged to cause rotation of a

stepless potentiometer used as a voltage divider, and its output can be monitored on a recorder. Moreover, the output of the potentiometer and a thermocouple can be recorded simultaneously to obtain accurate plots of needle motion *vs* temperature.

A few drops of the polymer sample are placed in an aluminum foil cup and rapidly cooled in liquid nitrogen to give a small button of glassy polymer. The foil cup is placed in the bottom of a small beaker which is full of methanol at a temperature below the anticipated glass transition temperature. A thermocouple junction is placed on the polymer button and the penetrometer needle is allowed to come into contact with the button at a point near the thermocouple. The methanol bath is allowed to increase in temperature while continuous records of needle position and temperature are made.

Below the penetration temperature, the record of the needle position is a straight line; when the penetration temperature is at hand, the needle suddenly begins to sink into the sample and the record exhibits an abrupt and continuous change in position until the needle stops at the bottom of the polymer bead. Meaningful values of penetration temperatures can be obtained only if the molecular weight of the prepolymers are low enough. In general, the functional dependence of penetration temperature and molecular weight is

$$T_p = A/\bar{M}_n + B$$

where \bar{M}_n is the molecular weight and A and B are constants.

(continued overleaf)

Note:

Requests for further information may be directed to:

Technology Utilization Officer
NASA Pasadena Office
4800 Oak Grove Drive
Pasadena, California 91103
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NASA has decided not to apply for a patent.

Source: James E. Potts, Jr. and
A. Clifford Ashcraft of
Union Carbide Corp./Chemicals and Plastics
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